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NASA TM X-2271

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REACTOR IRRADIATION OF THERMIONIC SHEATH INSULATORS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • MAY 1971

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA TM X-2271		

4. Title and Subtitle	5. Report Date
REACTOR IRRADIATION OF THERMIONIC SHEATH INSULATORS	May 1971
	6. Performing Organization Code

7. Author(s)	8. Performing Organization Report No.
John T. Mayer	E-5955
	10. Work Unit No. 120-27

9. Performing Organization Name and Address	11. Contract or Grant No.
Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135	

12. Sponsoring Agency Name and Address	13. Type of Report and Period Covered
National Aeronautics and Space Administration Washington, D. C. 20546	Technical Memorandum
	14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

Two beryllia and two alumina bonded trilayers (with niobium as the inner and outer layers) were irradiated at an average temperature of about 1200 K. The total fast fluence was between 2.3×10^{21} and 2.6×10^{21} neutrons/cm^2 (energy > 160 fJ (1 MeV)). None of the trilayers showed any gross changes in either the ceramic or in the metal-ceramic bond, although the alumina did show signs of grain boundary weakening. The sample diameters grew by 0.4 to 0.9 percent. Postirradiation ac resistance at 1275 K was higher than pretest values by as much as a factor of five.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Alumina Beryllia Thermionics Irradiation Insulators, ceramic	Unclassified - unlimited

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	22	$3.00

*For sale by the National Technical Information Service, Springfield, Virginia 22151

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SUMMARY

Thermionic sheath insulator specimens were irradiated to determine the effects of reactor irradiation at high temperature on the gross mechanical characteristics of the insulator and metal-insulator bond. Also of interest were changes in dimensions and electrical resistance.

Four specimens were irradiated (in the Engineering Test Reactor) at an average temperature of about 1200 K to a fast fluence of 2.3×10^{21} to 2.6×10^{21} neutrons per square centimeter (energy > 160 fJ (1 MeV)). The samples were cylindrical, three-layered, gas pressure-bonded structures with 0.38-millimeter niobium inner and outer layers. The ceramic layer was 0.25 millimeter thick. Two of the trilayers contained beryllia (BeO) and two contained alumina (Al_2O_3).

Postirradiation visual examination and metallography revealed no major cracks in the insulators and the bonds were generally good. Evidence of grain boundary weakening was seen in the alumina microstructure, however.

The outer diameter of the trilayers, nominally 1.62 centimeters, increased by 0.4 to 0.9 percent. The 1-kilohertz ac resistance was measured up to 1275 K in a vacuum furnace. The two alumina trilayers showed a three to fivefold increase in resistance due to irradiation over the entire temperature range (700 to 1275 K). One beryllia trilayer had a higher postirradiation resistance above 925 K; this increase was a factor of three at 1275 K. The other beryllia sample was evidently contaminated during the postirradiation heating period; this prevented any comparisons of the high-temperature resistance measurements.

INTRODUCTION

The characteristics of the sheath insulator are important factors in the performance of a thermionic space power system. In addition to minimizing electric conduction and preventing voltage breakdown, the insulator must provide a good heat conduction path and maintain dimensional stability. The thin layer of ceramic must perform these func-

tions over long time periods in a high-temperature environment (1000 K or more), and may operate in a cesium atmosphere as well.

Of particular concern for an in-pile diode are the effects of large neutron fluences on the insulator and metal-insulator bond. Some information does exist on radiation-induced changes in certain insulator properties (refs. 1 to 5), but very little testing of actual bonded structures has been done. For this reason, an irradiation test was performed on four cylindrical, fully bonded three-layered samples (metal-insulator-metal) with representative geometry. The primary objectives of the test were to determine the changes in the general mechanical condition of the insulator and the metal-insulator bond. Also of interest were changes in the insulator's microstructure and electrical resistivity.

The irradiation was conducted in the Engineering Test Reactor under an Oak Ridge National Laboratory (ORNL) program. The assistance of G. W. Keilholtz and D. A. Dyslin of ORNL during the irradiation phase is gratefully acknowledged. Post-irradiation examination was done in the Hot Laboratory of NASA's Plum Brook Station.

TEST SPECIMENS AND IRRADIATION ASSEMBLY

The four specimens tested are shown in figure 1, a photograph taken just before encapsulation. Each is a three-layered, fully bonded structure (trilayer) consisting of a ceramic layer in the center, and niobium layers on the inside and outside. The layer thicknesses are nominally 0.25 millimeter for the ceramic, and 0.38 millimeter for the niobium. The trilayers numbered 1 and 2 have a beryllia (BeO) ceramic layer, and numbers 4 and 5 have alumina (Al_2O_3).

The trilayers were fabricated by Thermo Electron Corporation using a gas-pressure bonding process. Impurities in the starting materials are shown in table I.

After preirradiation measurements of dimensions and resistance, the four trilayer specimens were assembled in two canisters as shown in figure 2. After the end caps were welded, the assemblies were evacuated overnight, backfilled with about 2.4 newtons per square centimeter (3.4 psia measured) of helium, and the fill tube welded shut. One canister (designated I) contained trilayers 2 and 5, the other (II) contained samples 1 and 4. After satisfactory leak testing, the canisters were sent to ORNL for assembly.

The ORNL irradiation assembly is shown in figure 3. Each canister was contained in a slip-fit capsule positioned inside an aluminum sleeve by locating tabs. Several such capsule-sleeve assemblies were stacked inside the stainless steel irradiation tube, which was cooled by reactor water flow on the outside. The capsule-sleeve gap contained a mixture of helium and argon. The proportions of this mixture (and thus the temperature drop across the gap) were controlled by a thermocouple inside the irradiation tube.

If a change in reactor conditions caused a change in this temperature, the gas mixture was adjusted to return the temperature to the normal condition. The capsule thermocouples were Chromel-Alumel. Flux wires ran along the length of the irradiation tube. More details on the irradiation assembly and helium-argon gas control system can be found in reference 6.

MEASURING APPARATUS AND PROCEDURE

Dimensions

Several measurements of the inner and outer diameters D_i and D_o and the length L of each trilayer were taken before irradiation using standard micrometers. Postirradiation values of D_o and L were measured with an optical gage and are accurate to 0.0025 millimeter; the dimensions of a control trilayer were within 0.025 millimeter (0.001 in. measured) of previous measurements. The postirradiation value of D_i was measured only once, with a modified inside micrometer, and is probably not as accurate.

Resistance Measurements

The electrical resistance of each trilayer was measured up to 1275 K before and after irradiation.

Apparatus. - Figure 4 shows the vacuum furnace used in preirradiation measurements. Its element is resistance-heated using a dc power supply. A diffusion pump vacuum system maintained pressure between 3.0×10^{-4} and 7.0×10^{-4} newtons per square meter (2.0×10^{-6} to 5.0×10^{-6} torr measured). The trilayer was suspended in the heat zone from above by means of the fixture shown in figure 4. Electrical contact with each metal layer was made through the niobium spacers, while the alumina spacers insulated the clamp from the opposite layer. Temperature was measured with a Chromel-Alumel thermocouple whose junction was on the axis within the trilayer. The two rods and thermocouple were brought out through insulating fittings in the top cover plate.

The furnace and vacuum system used in postirradiation measurements was essentially the same, except that it had a tungsten mesh heating element which was ac (60 Hz) resistance-heated. Also, since it was difficult to put the thermocouple junction within the trilayer, as in the preirradiation measurements, it was brought in from the side. A calibration run with thermocouples in both locations showed that the maximum difference was 4 K. Figure 5 shows the fixture used to hold the trilayers for postirradiation resistance measurements. The trilayer contacts (tungsten strip and wire)

were bent so spring tension held them against the trilayer. The two contacts on each niobium layer provided some redundancy. The ceramic plate material was boron nitride (BN) in the first run (trilayer 5). However, the current leakage through it was somewhat large above 1200 K, so alumina was used for the other runs.

Calibration runs on both fixtures were made to measure their resistance. This was required in order to calculate the trilayer resistance from the measured resistance. As indicated previously, the BN plate had a fairly small resistance above 1200 K, so the calculated postirradiation resistance for sample 5 in this temperature range has a larger error (say 10 or 20 percent). Other than this, the resistance of both fixtures was greater than 20 times that of the trilayer resistance, so the calculated values are quite accurate.

Procedure. - Before the preirradiation resistance measurements were taken, all trilayers were sandblasted with Al_2O_3 to remove any metallic particles from the exposed insulator surfaces. This did not change the room-temperature resistivity of any of the trilayers. Sandblasting was required for some of the samples during postirradiation operations. This was evidently due to contamination of the exposed insulator surfaces during the first heating in the furnace. Sandblasting with alumina powder corrected this condition for samples 2 and 4, but only partially helped with sample 1.

The ac resistance measurements were taken using simple series and parallel capacitance comparison bridges at 1 kilohertz. The procedure was to heat the trilayer to 1275 K, hold it at that temperature until the resistance stabilized, and take resistance measurements at various temperatures during cooldown. The temperature was allowed to stabilize for each datum point.

IRRADIATION HISTORY AND DISASSEMBLY

The irradiation tube (ORNL experiment 41-40) was inserted in the southwest quadrant of the J-12 core filler piece of the Engineering Test Reactor (ETR). It was irradiated for five reactor cycles (numbers 97 to 101) between July 30, 1968 and April 26, 1969.

The specimen temperature varied over a wide range (1075 to 1475 K), often with large differences on successive days. This was primarily due to difficulties encountered with the helium-argon gas system. Most of the temperature data were in the range 1075 to 1275 K, however; the overall average temperature was about 1200 K.

From the ETR cycle reports it is estimated that the trilayers underwent about 75 thermal cycles between ambient and operating temperature. All of the temperature drops were essentially scram-type reductions.

From capsule monitors, the fast neutron fluences (energy $>$ 160 fJ (1 MeV)) were as follows: canister I, 2.3×10^{21} neutrons per square centimeter; II, 2.6×10^{21} neutrons

per square centimeter. These correspond to average fast neutron fluxes of 1.6×10^{14} and 1.8×10^{14} neutrons per square centimeter per second, respectively.

Los Alamos investigators (refs. 4 and 5) have recently proposed that comparison of radiation effects from different reactors is more valid if the fast neutron fluence includes energies > 16 femtojoules (0.1 MeV). To convert the fluences in the present report to a fluence > 16 femtojoules, they should be multiplied by about two.

Data from monitors in the core filler piece were used to estimate the average thermal neutron flux at 2.3×10^{14} neutrons per square centimeter per second; the calculated thermal neutron fluence was 3.3×10^{21} neutrons per square centimeter.

The gamma heating rate was estimated at 13 watts per gram, although no actual data were taken during the irradiation.

After removal from the ETR, the irradiation tube was shipped to ORNL for partial disassembly. The two capsule-canister assemblies were then sent to NASA's Plum Brook Reactor Facility for further disassembly and examination.

Some difficulty was encountered in removing the trilayers from the canisters. This was due to the strong adhesion between all the close-fitting stainless steel pieces. The trilayers were somewhat nicked and scraped during disassembly, but it is felt that this did not affect the subsequent measurements.

RESULTS AND DISCUSSION

Gas Analysis

Prior to disassembly, the gas inside canister II was subjected to chromatographic analysis (canister I was inadvertently cut into during ORNL disassembly). The tests showed that the internal atmosphere had increased from 2.4 to about 5.9 newtons per square centimeter (3.4 to 8.4 psia measured). The gas increase consisted of about 12 percent nitrogen, 2 percent oxygen, <0.5 percent argon, and the balance helium. This could have resulted from a very small leak which admitted helium and argon during the irradiation, and air during the postirradiation period.

Visual Examination

Figure 6 shows the four trilayers after removal from the canisters. The appearance of the niobium is similar to that before irradiation except for sample 2, which looks somewhat corroded. The visible part of the insulator darkened noticeably for samples 1 and 4 (compare with fig. 1). The insulator defect on sample 1 (visible at the

upper left in fig. 6) was there before irradiation.

Each of the two BeO samples had about six cracks in the inner niobium at the stepped end. Some of these can be seen in figure 6. It is possible that thermal expansion of the canister and cap, or disassembly operations, caused these. None of these cracks extend beyond the visible portion of the BeO, and no such cracks were observed at any other location.

Examination of the face of the ceramic at the flat end of the trilayers showed a difference between the two materials. The two BeO samples were in fairly good condition, except for a shallow depression along the outer niobium boundary (see fig. 7(a)). Most of the BeO and niobium interface looked quite good, as in figure 7(b). The radial crack in this figure was also quite shallow.

Both Al_2O_3 samples, on the other hand, had thin cracks in the body of the insulator at the flat end of the trilayer (see fig. 7(c)). These cracks extended 50 to 75 percent around the circumference and had a sharper appearance than the BeO depressions. It should be pointed out, however, that the cracks were so small that they could have gone unnoticed during preirradiation examinations. In addition to the thin crack, trilayer 5 had a wider, short crack (see fig. 7(d)) and a significant portion of alumina-niobium bond separation. However, this was probably caused or at least intensified by a bolt striking the trilayer inside diameter during removal from the canister. The resulting nick can be seen in figure 7(d), near a chip in the Al_2O_3 which exposed the lighter material beneath. Figures 7(c) and (d) do show, however, that the alumina-niobium bond held up well.

The extent of the insulator cracks and separations will be discussed further in the Metallography section.

Metallography

Two metallographic sections of each trilayer were prepared. One was a transverse (circular) section perpendicular to the axis at the center. The other was a longitudinal section parallel to the axis, extending from the center of the trilayer to the flat end. All sections were examined and photographed in the as-polished condition.

Although the transverse sections remained intact, three of the four longitudinal sections broke during the cutting operations. For alumina sample 5, the break occurred in the body of the insulator only, while samples 1 and 4 had breaks in the niobium as well. However, at least one-half of each longitudinal section remained intact for examination. These sections showed some of the circumferential cracks and depressions at the flat end that were seen in the earlier examinations. However, the furthest extent of these into the body of the insulator was about 1 millimeter.

Photomicrographs of typical transverse sections of each trilayer are shown in figures 8 and 9. The beryllia sections (fig. 8) show less grain pullout (from grinding operations) than the alumina sections (fig. 9). Sample 4 is particularly bad in this respect. The amount of pullout is usually a good measure of the degree of grain boundary weakening and/or separation.

The niobium-insulator bond seems to be physically sound in most trilayer sections. Figures 8 and 9 show what looks like a separation, but this is actually only a small V-shaped depression. By focusing down on the section, especially at higher magnification, the bond could be seen. The appearance of the bond was better in the earlier stages of polishing, as is shown in figure 10 for trilayer 1. It is likely that the final polishing caused the depression at the bond. Trilayer 5 had a very real separation between the insulator and inner niobium over about 25 percent of the circumference of the transverse section. The maximum gap was about 0.030 millimeter. It is possible that this separation was caused by the disassembly damage mentioned earlier.

Trilayers 2 and 4 (figs. 8(b) and 9(a)) show some kind of reaction layer at the niobium-insulator bond. This interaction was seen in all sections of these two samples only. Better detail of the sample 2 reaction is shown in figure 11, a photomicrograph taken before final polishing. It is possible that this reaction was a result of fabrication due to a metallizing layer often used to enhance bonding. (Private communication from V. Poirier, Thermo Electron Corporation.) This theory is supported by figure 12, a photomicrograph of trilayer 4 after irradiation. The figure shows an area of the transverse section where niobium has "extruded" into a crack in the alumina. A second grinding operation showed this crack to be at least 3 millimeters in length. No reaction layer is seen along these niobium "fingers", but only at the original interface where the postulated coating would have been. No explanation is available as to why no reaction layer is seen in the other two trilayers (1 and 5).

The radial cracks seen in figures 8(b) and 11 are quite typical of trilayer 2; it had about 100 such cracks in the transverse section and many in the longitudinal. Not all cracks extended all the way from layer to layer, but several were associated with small cracks in the niobium as shown in the figures. Sample 1 showed considerably fewer cracks. Again, it is difficult to determine whether these cracks were present before irradiation.

Dimensional Measurements

Preirradiation and postirradiation dimensions of the trilayers are shown in table II. Table II also shows the difference between postirradiation and preirradiation values of D_o, D_i, and L. Some of this growth may possibly be due to heat treatment alone. An

alumina control trilayer which was held at 1325 K for over 3000 hours (out of pile) was found to have grown about 0.04 millimeter (0.0015 in. measured) in all three dimensions. This may be due to some kind of relaxation process in the niobium. Despite this, there seems to be a definite radiation-induced growth which is greater for the trilayers in canister I (samples 2 and 5).

Resistance Measurements

A check of room-temperature dc resistance of each trilayer was made prior to the high-temperature measurements; all were greater than 10^7 ohms (the limit of the bridge).

Preirradiation and postirradiation resistance values as a function of temperature are shown in figure 13.

For BeO sample 1 (fig. 13(a)), anomalous behavior in the preirradiation measurements made it difficult to draw any conclusions about the data. Note, however, that at low temperatures during the first warmup, the postirradiation resistance showed an increase over preirradiation values. After the postulated contamination occurred (during the first warmup), the postirradiation resistance was lower. Sandblasting produced only a slight increase.

BeO sample 2 showed an increase over preirradiation values only for temperatures greater than 925 K (fig. 13(b)). At 1275 K, this increase was about a factor of three.

The two alumina trilayers (figs. 13(c) and (d)) are quite similar in that both showed a three to sixfold increase in resistance which was fairly uniform from about 700 to 1275 K. This increase is in general agreement with the results of reference 7, in which all of eight alumina specimens tested showed a five to tenfold increase in resistivity. Irradiation temperatures in the reference 7 test were 1075 to 1275 K, and the fast fluence was about 1.5×10^{20} neutrons per square centimeter (energy > 160 fJ (1 MeV)). Recent Los Alamos tests on alumina (ref. 4) showed just the opposite; all specimens decreased in resistivity after irradiation. Temperature in this case was about 1000 K; fluences ranged from about 0.2×10^{21} to 1.1×10^{21} neutrons per square centimeter (energy > 160 fJ (1 MeV)). The fast fluence can also be expressed as 3.6×10^{21} to 9.8×10^{21} neutrons per square centimeter with energies > 16 femtojoules (0.1 MeV).

SUMMARY OF RESULTS

Four niobium sheath insulator specimens (two with beryllia, two with alumina) were irradiated at an average temperature of about 1200 K to a fast fluence of 2.3×10^{21} to 2.6×10^{21} neutrons per square centimeter (energy > 160 fJ (1 MeV)).

Postirradiation examination revealed no major damage to the insulators or metal-insulator bonds. An interaction layer was seen at the boundaries in two of the specimens (one of each type), but there was evidence that this was a result of fabrication. The alumina microstructure showed a significant amount of pullout, indicating a weakening of the grain boundaries. The beryllia showed much less pullout.

Increases in sample diameters and lengths varied from 0.4 to 0.9 percent, with no discernible pattern. The ac (1000-Hz) resistance of three of the trilayers increased two to fivefold over preirradiation values at high temperatures. One of the beryllia samples had a lower resistance, which was probably due to contamination during the heating cycle of the resistance measurements.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 20, 1971,
120-27.

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TABLE I. - STARTING MATERIAL IMPURITIES[a]

Impurity	Niobium[b]	Beryllia[c]	Alumina[d]
	Concentration, ppm		
Aluminum	10	115	------
Boron	---	2	10
Carbon	35	----	------
Calcium	<10	50	------
Calcia	---	----	10 500
Cobalt	<10	<1	------
Chromium	<10	7	40
Copper	<10	<1	5
Iron	80	80	200
Gallium	---	----	40
Hydrogen	7	----	------
Lithium	---	3.5	------
Magnesium	<10	1200	50
Manganese	<10	12	Trace
Molybdenum	[e]<10	----	------
Nitrogen	10	----	------
Sodium	(e)	75	------
Nickel	20	5	------
Oxygen	75	----	------
Silicon	30	125	------
Silica	---	----	13 500
Tin	[e]<10	----	------
Tantalum	550	----	------
Titanium	<10	<2	40
Zinc	---	120	------
Zirconium	<10	----	------

[a]Vendor's analyses.

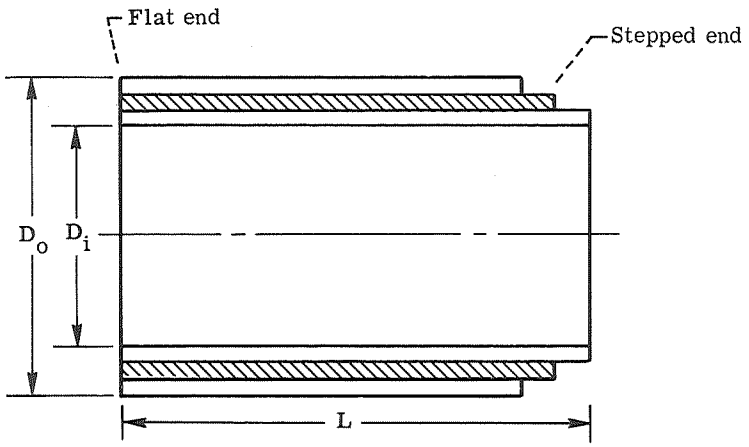
[b]Chemical analysis, quoted as "typical."

[c]Certified analysis.

[d]Silica and calcia by wet analysis; others by spectrographic analysis. Analysis quoted as "typical."

[e]Not detected.

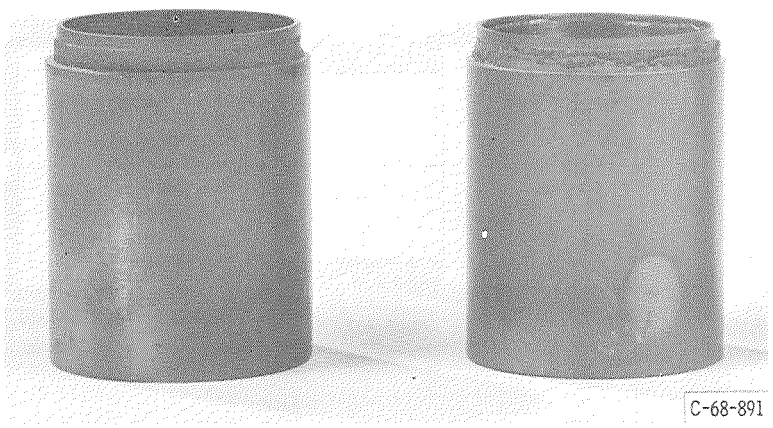
TABLE II. - TRILAYER DIMENSIONS BEFORE AND AFTER IRRADIATION



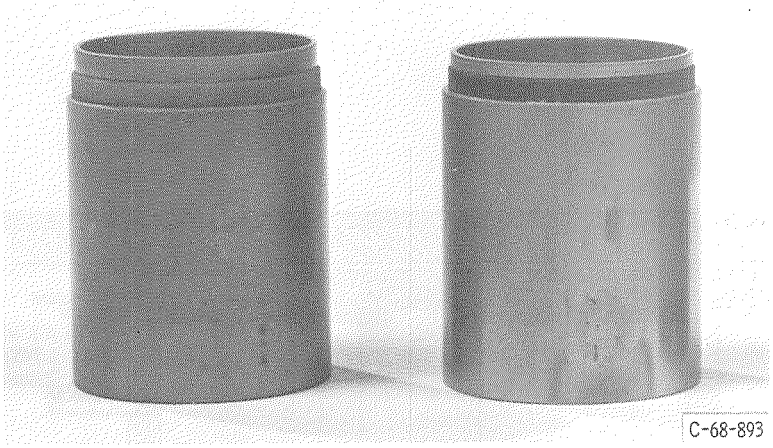
Specimen	Neutron fluence,[a] $\frac{\text{neutrons}}{\text{cm}^2}$	Outer diameter, D_o	Inner diameter, D_i	Length, L	Outer diameter, D_o	Inner diameter, D_i	Length, L
		Mean dimensions, cm (in.)[b]			Increase, mm (mils)		
1 (Beryllia)	0	1.632 (0.6425)	1.435 (0.5651)	2.103 (0.8280)	0.063 (2.5)	0.081 (3.2)	0.117 (4.7)
	2.6×10^{21}	1.638 (0.6450)	1.444 (0.5683)	2.115 (0.8327)			
2 (Beryllia)	0	1.621 (0.6382)	1.432 (0.5636)	2.090 (0.8227)	0.145 (5.7)	0.112 (4.4)	0.188 (7.4)
	2.3×10^{21}	1.634 (0.6439)	1.443 (0.5680)	2.109 (0.8301)			
4 (Alumina)	0	1.618 (0.6372)	1.431 (0.5633)	2.131 (0.8391)	0.079 (3.1)	0.132 (5.2)	0.053 (2.1)
	2.6×10^{21}	1.626 (0.6403)	1.444 (0.5685)	2.137 (0.8412)			
5 (Alumina)	0	1.633 (0.6428)	1.438 (0.5663)	2.096 (0.8251)	0.132 (5.2)	0.056 (2.2)	0.163 (6.4)
	2.3×10^{21}	1.646 (0.6480)	1.440 (0.5685)	2.112 (0.8315)			

[a]Neutron energy > 160 fJ (1 MeV)

[b]Actual measured dimensions (i.e., in.) are in parentheses.



Specimen 1 (BeO). Specimen 2 (BeO).



Specimen 4 (Al_2O_3). Specimen 5 (Al_2O_3).

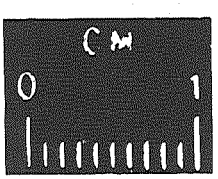


Figure 1. - Trilayer test specimens before irradiation.

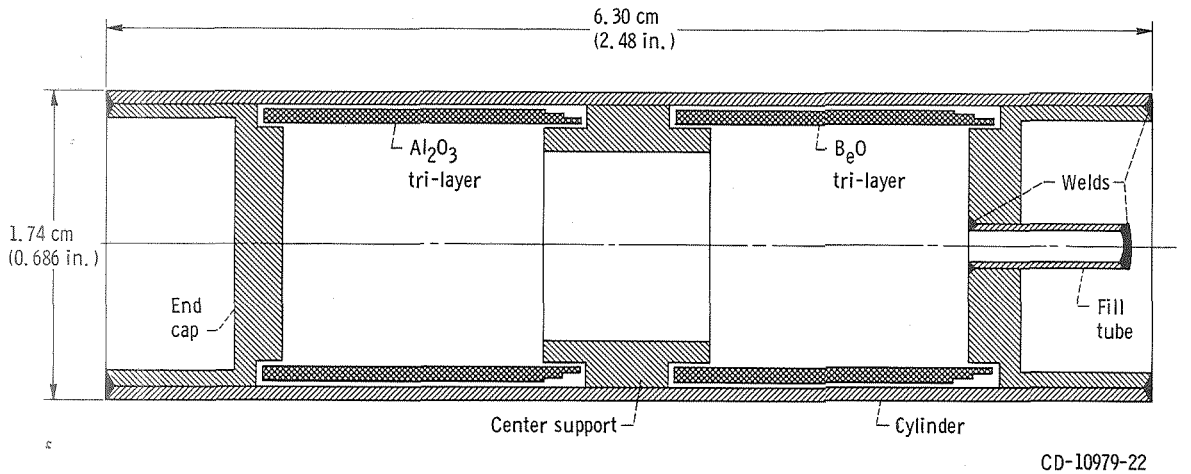


Figure 2. - Tri-layer - canister assembly. (All canister parts of AISI 347 stainless steel.)

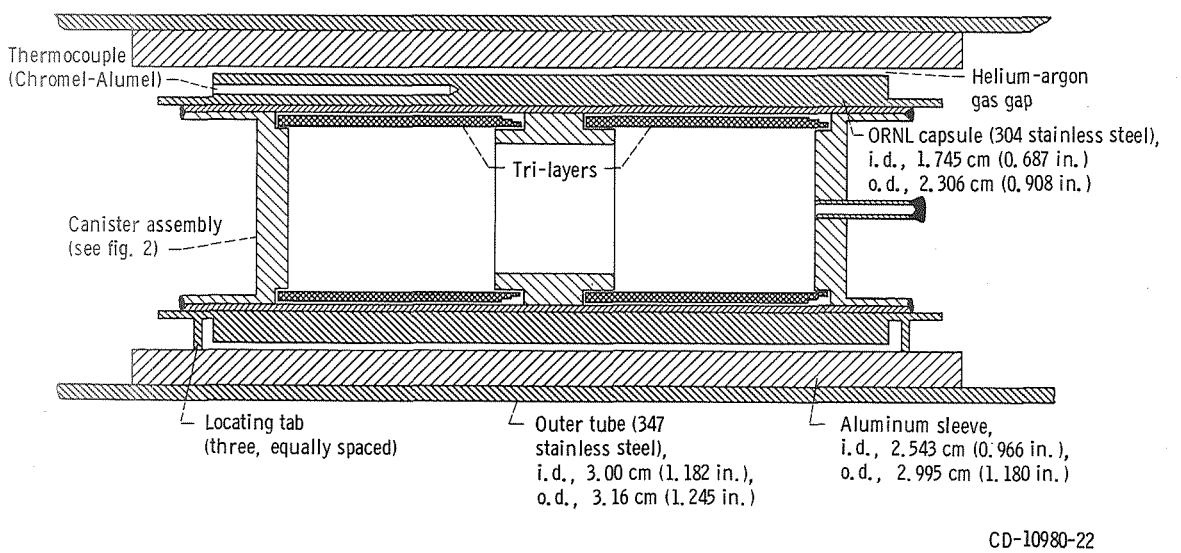


Figure 3. - ORNL irradiation assembly.

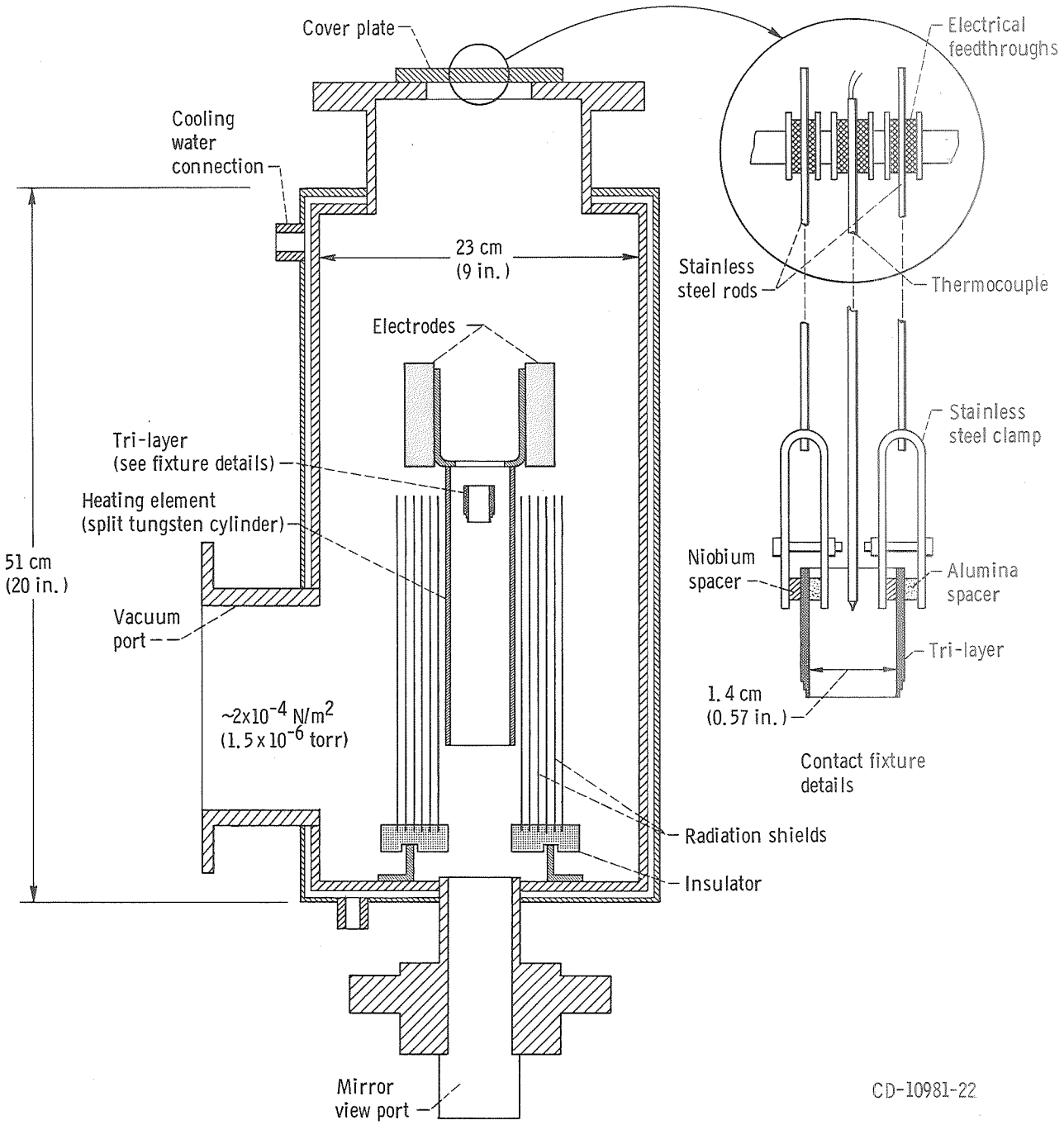


Figure 4. - Vacuum furnace and contact fixture used in pre-irradiation resistance measurements.

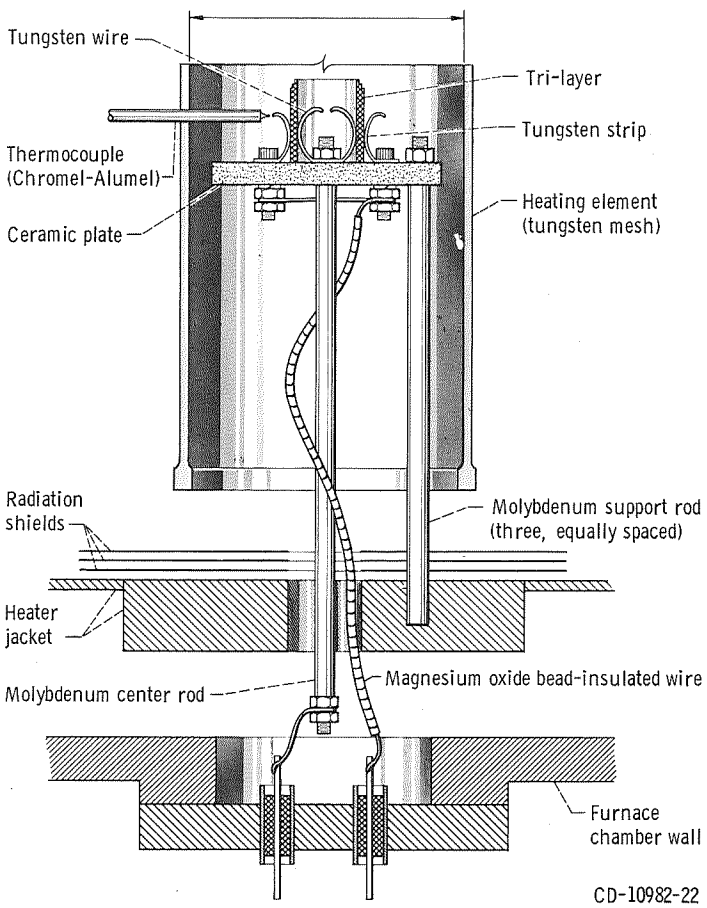
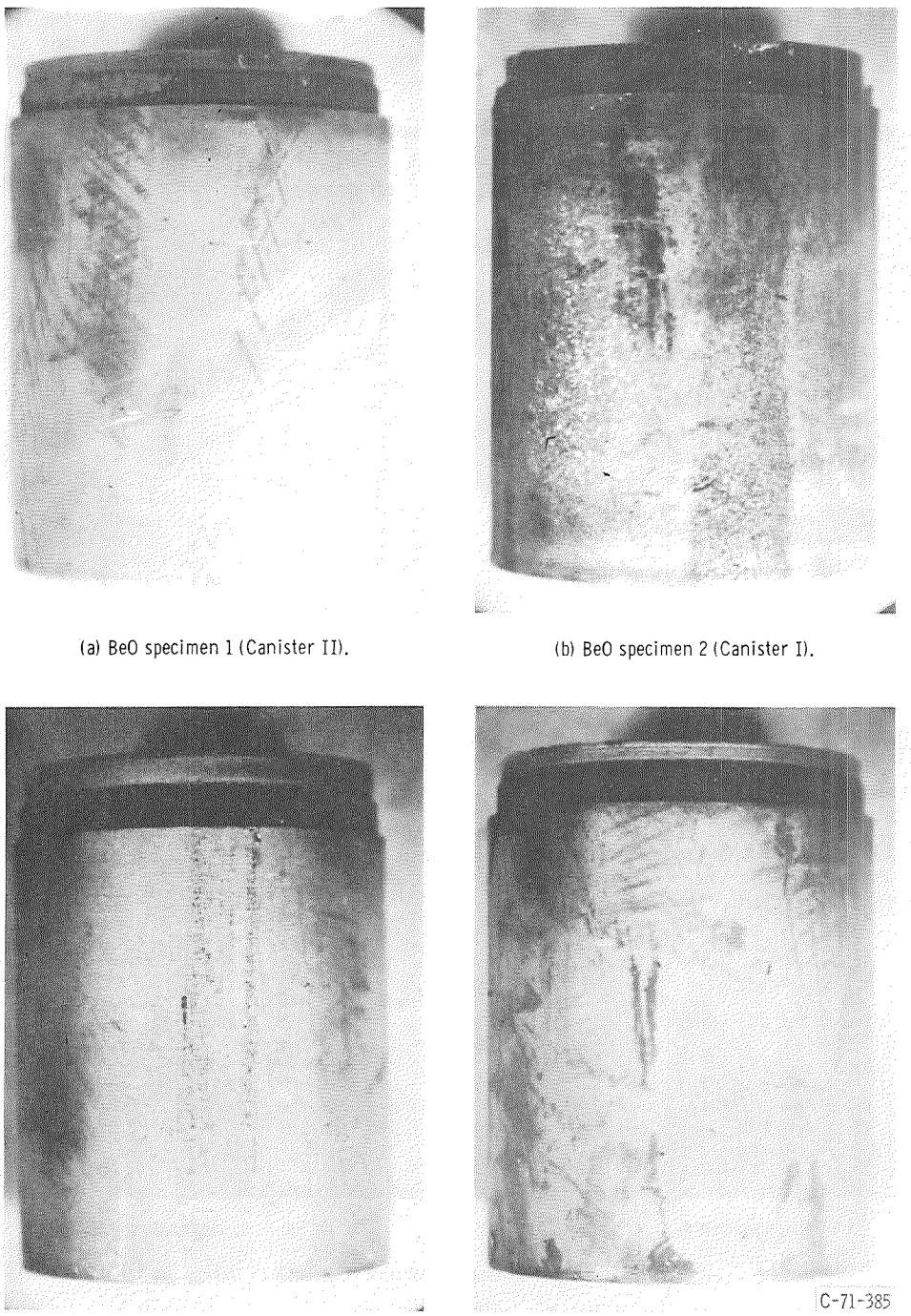


Figure 5. - Furnace heat zone and contact fixture used in post-irradiation resistance measurements.



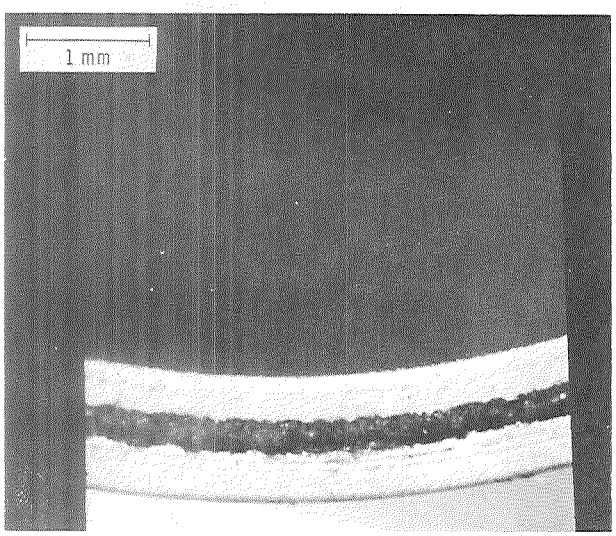
(a) BeO specimen 1 (Canister II).

(b) BeO specimen 2 (Canister I).

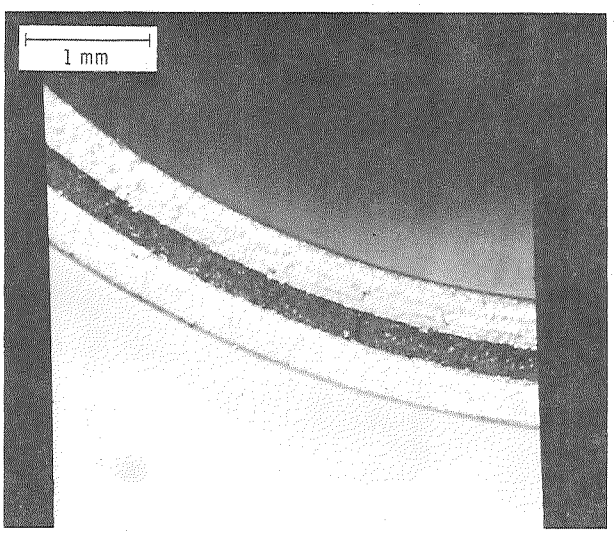
(c) Al_2O_3 specimen 4 (Canister II).

(d) Al_2O_3 specimen 5 (Canister I).

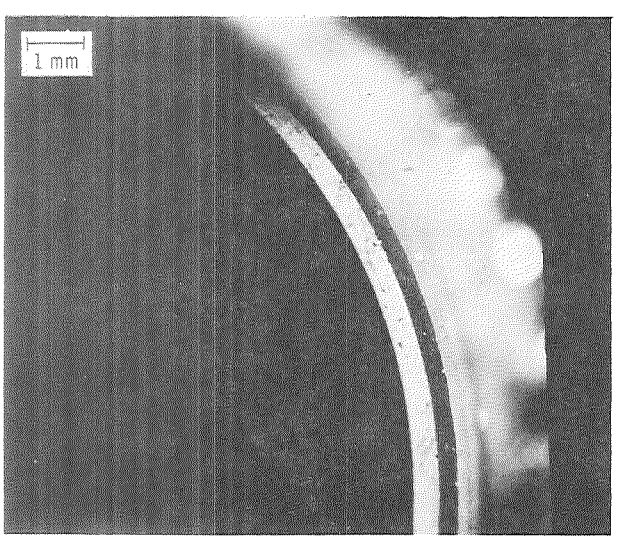
Figure 6. - General appearance of trilayers after irradiation.



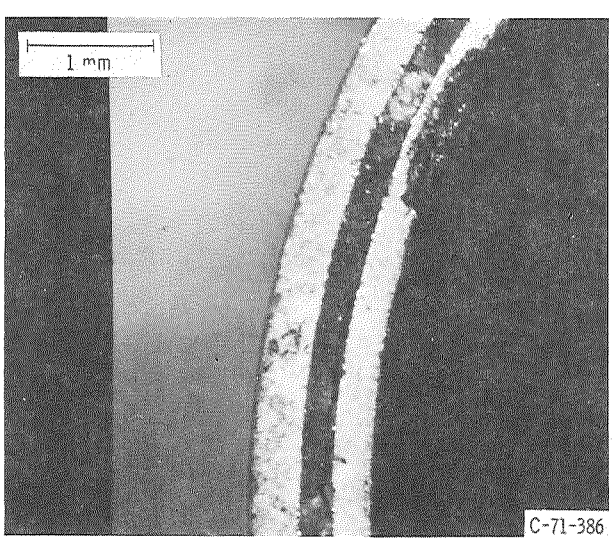
(a) BeO specimen 1.



(b) BeO specimen 1.

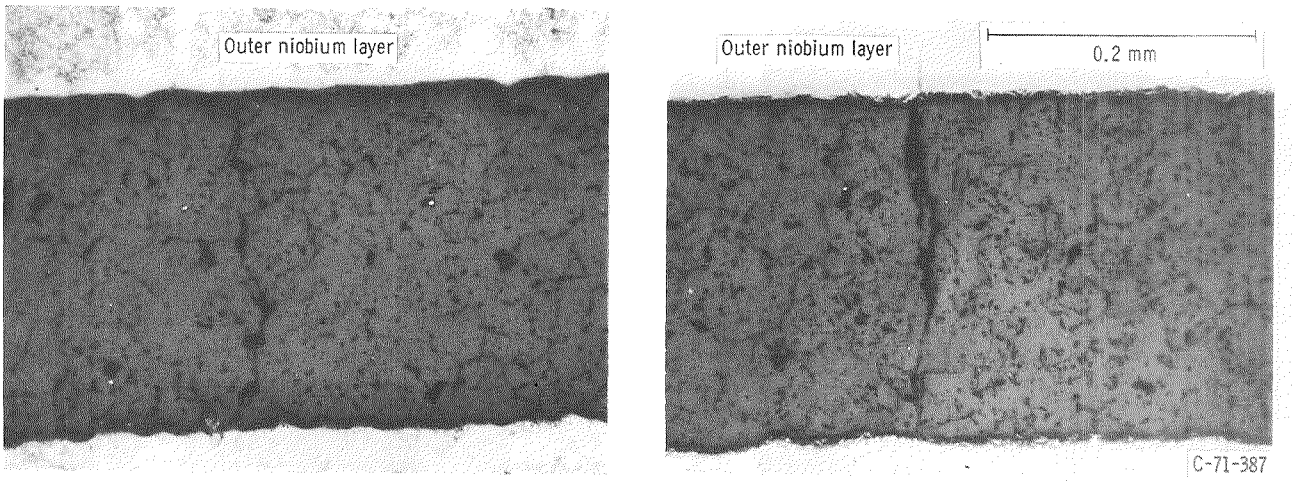


(c) Al_2O_3 specimen 4.



(d) Al_2O_3 specimen 5.

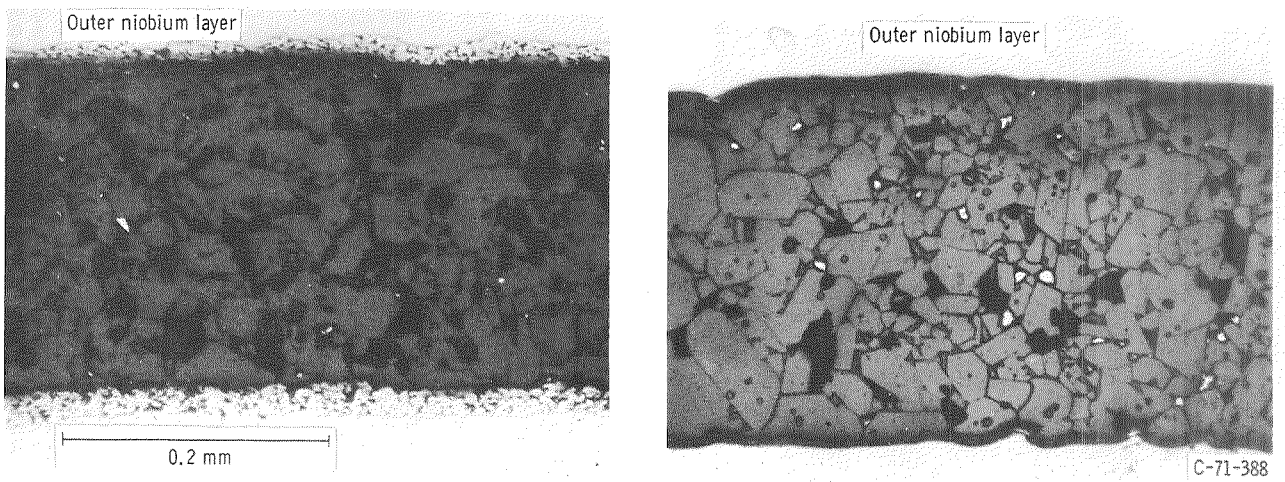
Figure 7. - Trilayer appearance at flat end.



(a) BeO sample 1.

(b) BeO sample 2.

Figure 8. - Photomicrographs of beryllia trilayers (transverse sections) after irradiation (X250, unetched).



(a) Al_2O_3 sample 4.

(b) Al_2O_3 sample 5.

Figure 9. - Photomicrographs of alumina trilayers (transverse sections) after irradiation (X250, unetched).

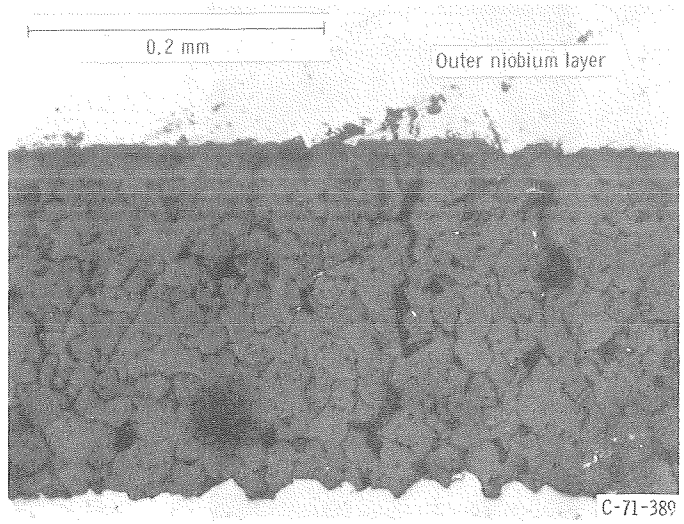


Figure 10. - Photomicrograph of beryllia trilayer 1 (transverse section) before final polish (X250, unetched).

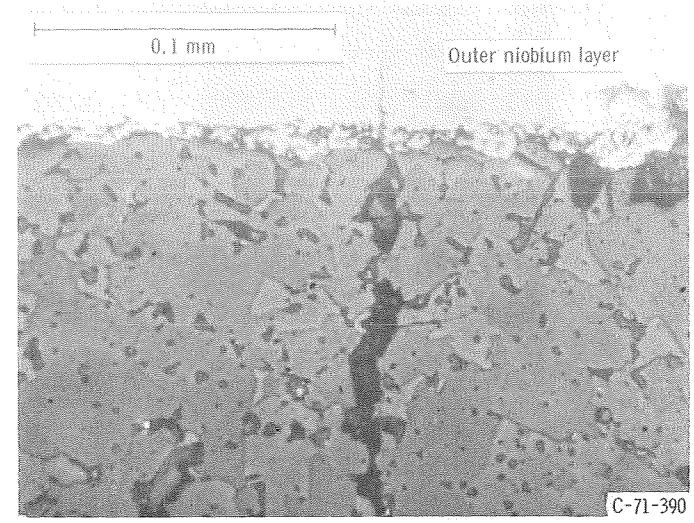


Figure 11. - Photomicrograph of beryllia trilayer 2 (longitudinal section) before final polish (X500, unetched).

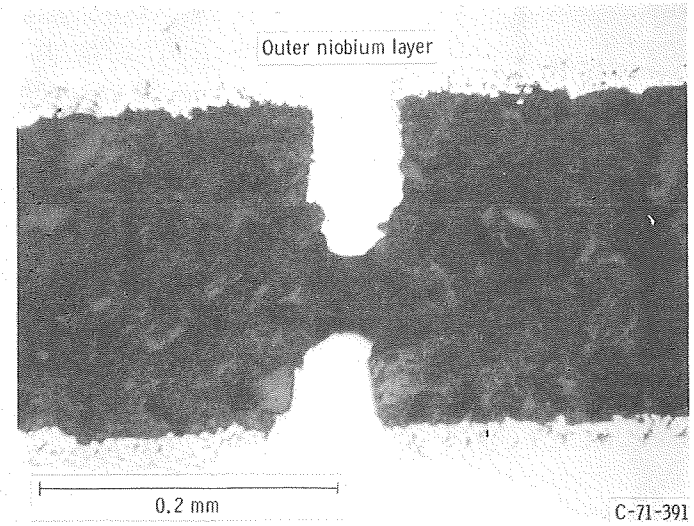


Figure 12. - Photomicrograph of alumina trilayer 4 (transverse section) after irradiation (X250, unetched).

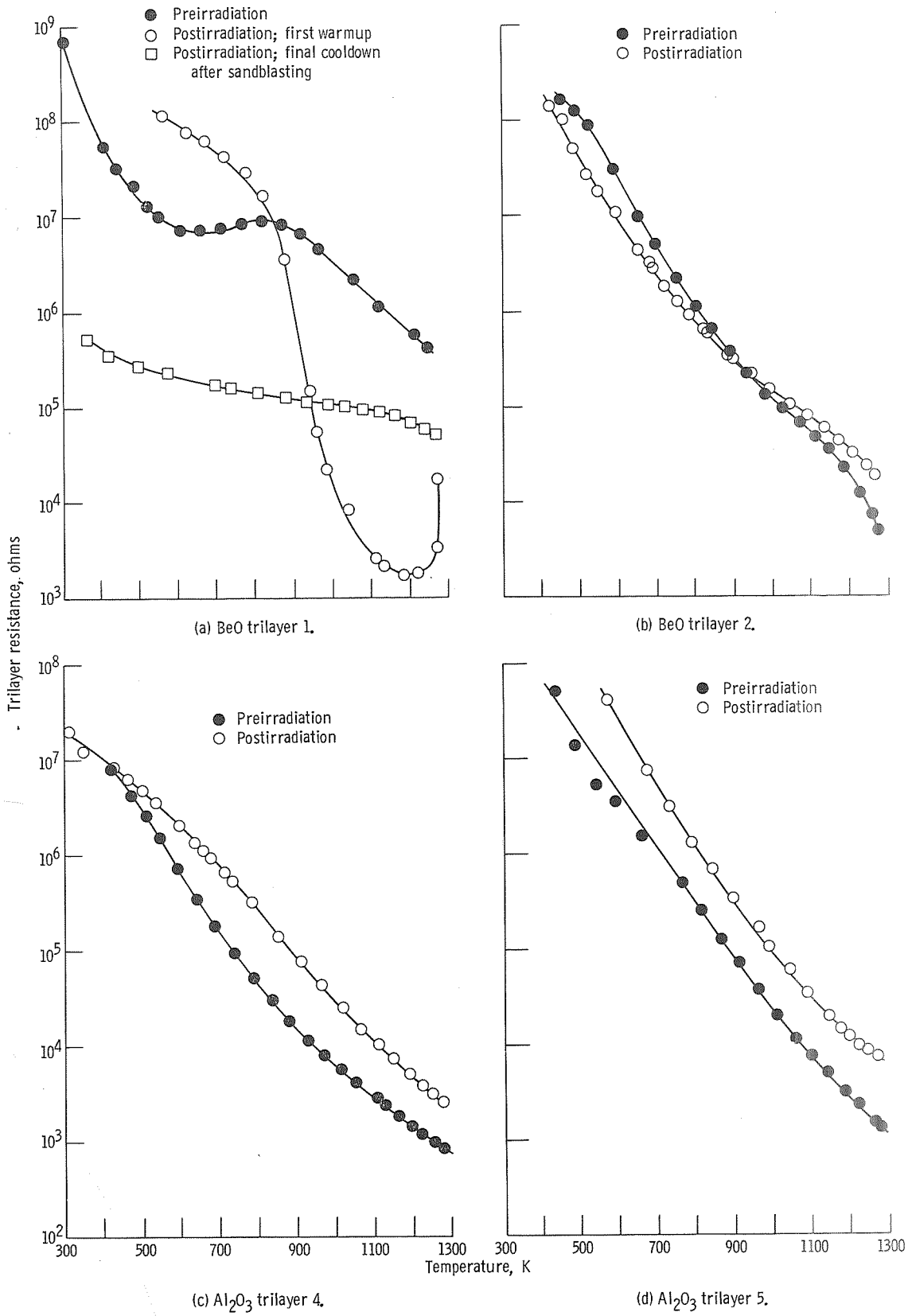


Figure 13. - Preirradiation and postirradiation resistance of various trilayers.

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